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Tool with tool body and protective layer system

This description has an Appendix A.

The present invention is directed on a tool with a tool body and a wear resistant layer system, wherein the layer system comprises at least one layer of MeX, wherein

- Me comprises titanium and aluminum,
- X is at least one of nitrogen and of carbon.

<u>Definition:</u>

• The term Q_I is defined as the ratio of the diffraction intensities I(200) to I(111), assigned respectively to the (200) and (111) plains in the X-ray diffraction of a material using the θ -2 θ method. Thus, there is valid Q_I = I(200)/I(111). The intensity values were measured with the following equipment and with the following settings:

15 Siemens Diffractometer D500

Power:

Operating voltage: 30 kV

Operating current: 25 mA

Aperture Diaphragms:

Diaphragm position I: 1'

Diaphragm position II: 0.1°

20 Detector Diaphragm:

Soller slit

Time constant:

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29 angular speed:

0.05°/min

Radiation:

Cu-Kα(0.15406 rm)

When we refer to "measured according to MS" we refer to this equipment and to these settings. Thereby, all quantitative re-

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sults for Q_{I} and I throughout this application have been measured by MS.

- . We understand by "tool body" the uncoated tool.
- We understand under "hard material" a material with which tools which are mechanically and thermally highly loaded in operation are coated for wear resistance. Preferred examples of such materials are referred to below as MeX materials.

It is well-known in the tool-protecting art to provide wear resistant layer systems which comprise at least one layer of a hard material, as defined by MeX.

The present invention has the object of significantly improving the lifetime of such tools. This is resolved by selecting for said at least one layer a Q_1 value, for which there is valid

 $Q_x \ge 1$

- and wherein the tool body is made of high speed steel (HSS) or of cemented carbide, whereby said tool is not a solid carbide end mill or a solid carbide ball nose mill. Further, the value of I(200) is higher by a factor of at least 20 than the intensity noise average level as measured according to MS.
- According to the present invention it has been recognised that the Q_1 values as specified lead to an astonishingly high improvement of wear resistance, and thus of lifetime of a tool, if such a tool is of the kind as specified.
- Up to now, application of a wear resistant layer systems of MeX hard material was done irrespective of interaction between tool body material and the mechanical and thermal load the tool is

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subjected to in operation. The present invention thus resides on the fact that it has been recognised that an astonishing improvement of wear resistance is realised when selectively combining the specified Q₁ value with the specified kind of tools, thereby realising a value of I(200) higher by a factor of at least 20 than the average noise intensity level, both measured with MS.

With respect to inventively coating cemented carbide tool bodies, it has further been recognised that a significant improvement in lifetime is reached if such cemented carbide tools are inserts, drills or gear cutting tools, as e.g. hobs or shaper cutters, whereby the improvement is especially pronounced for such inserts or drills.

The inventively reached improvement is even increased if Q_i is selected to be at least 2, and an even further improvement is realised by selecting Q_i to be at least 5. The largest improvements are reached if Q_i is at least 10. It must be stated that Q_i may increase towards infinite, if the layer material is realised with a unique crystal orientation according to a diffraction intensity I(200) at a vanishing diffraction intensity I(111). Therefore, there is not set any upper limit for Q_i which is only set by practicability.

As is known to the skilled artisan, there exists a correlation between hardness of a layer and stress therein. The higher the 25 stress, the higher the hardness.

Nevertheless, with rising stress, the adhesion to the tool body tends to diminish. For the tool according to the present invention, a high adhesion is rather more important than the highest possible hardness. Therefore, the stress in the MeX layer is

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advantageously selected rather at the lower end of the stress range given below.

These considerations limit in practice the Q_{τ} value exploitable.

- In a preferred embodiment of the inventive tool, the MeX material of the tool is titanium aluminum nitride, titanium aluminum num carbonitride or titanium aluminum boron nitride, whereby the two materials first mentioned are today preferred over titanium aluminum boron nitride.
 - In a further form of realisation of the inventive tool, Me of the layer material MeX may additionally comprise at least one of the elements boron, zirconium, hafnium, yttrium, silicon, tungsten, chromium, whereby, out of this group, it is preferred to use yttrium and/or silicon and/or boron. Such additional element to titanium and aluminum is introduced in the layer material, preferably with a content i, for which there is valid

0.05 at. $% \le i \le 60$ at.%,

taken Me as 100 att.

A still further improvement in all different embodiments of the 20 at least one MeX layer is reached by introducing an additional layer of titanium nitride between the MeX layer and the tool body with a thickness d, for which there is valid

 $0.05 \ \mu m \le d \le 5 \ \mu m$.

In view of the general object of the present invention, which
is to propose the inventive tool to be manufacturable at lowest
possible costs and thus most economically, there is further

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proposed that the tool has only one MeX material layer and the additional layer which is deposited between the MeX layer and the tool body.

Further, the stress σ in the MeX is preferably selected to be

1 GPa ≤ σ ≤ 4 GPa, thereby most preferably

within the range

1.5 GPa $\leq \sigma \leq$ 2.5 GPa .

The content x of titanium in the Me component of the MeX layer is preferably selected to be

70 at $\$ \ge x \ge 40$ at \$, thereby in a further

preferred embodiment within the range

65 at $% \ge x \ge 55$ at % .

On the other hand, the content y of aluminum in the Me component of the MeX material is preferably selected to be

30 at $% \le y \le 60$ at % = 100, in a further preferred

embodiment even to be

35 at $% \le y \le 45$ at % ...

In a still further preferred embodiment, both these ranges, i.e. with respect to titanium and with respect to aluminum are fulfilled.

The deposition, especially of the MeX layer, may be done by any known vacuum deposition technique, especially by a reactive PVD coating technique, as e.g. reactive cathodic arc evaporation or

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reactive sputtering. By appropriately controlling the process parameters, which influence the growth of the coating, the inventively exploited Q_i range is realised.

To achieve excellent and reproducible adhesion of the layers to the tool body a plasma etching technology was used, as a preparatory step, based on an Argon plasma as described in Appendix A, which document is integrated to this description by reference, with respect to such etching and subsequent coating. This document accords with the US application No. 08/710 095 of the same inventor (two inventors!) and applicant as the present application.

Examples 1

An arc ion plating apparatus using magnetically controlled arc sources as described in Appendix A was used operated as shown in table 1 to deposit the MeX layer as also stated in table 1 on cemented carbide inserts. The thickness of the MeX layer deposited was always 5 µm. Thereby, in the samples Nr. 1 to 7, the inventively stated Q₁ values where realised, whereas, for comparison, in the samples number 8 to 12 this condition was not fulfilled. The I(200) value was always significantly larger than 20 times the noise average value, measured according to MS. The coated inserts were used for milling under the following conditions to find the milling distance attainable up to delamination. The resulting milling distance according to the lifetime of such tools is also shown in table 1.

Test cutting conditions:

- Material being cut: SKD 61 (HRC45)

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- Cutting speed:

100 m/min

- Feed speed:

0.1 m/edge

- Depth of cut:

2 mm

The shape of the inserts coated and tested was in accordance with SER 42 TN (G9).

It is clearly recognisable from table 1 that the inserts, coated according to the present invention, are significantly more protected against delamination than the inserts coated according to the comparison conditions.

10 Further, the result of sample 7 clearly shows that here the stress and thus hardness of the layer was reduced, leading to lower cutting distance than would be expected for a high Q; of 22.5, still fulfilling the stress-requirements as defined above.

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(Table 1)

Sample No.			Coating Conditions	Lona	Layer			1 (200) /	Residual Stress GPa	Attainable Cutting Dis-	
		84 87 87 87 87 88 88 88 88 88 88 88 88 88		Arc		×	~	I(111)		tance (m) (distance	Remarks
		Voltage	Voltage N, pressure	Current						mation)	
		(-V)	(nbar)	(A)							
	٢	60	2.0 x 10-7	150	(T1,A1,)N	0.5	0.5	1.5	5.2	2.2m (2.1m)	
	2	60	9.0 x 10.7	150	(T1,A1,) N	0.5	0.5	6.7	4.80	2.8m (2.5m)	
Present	ယ	ŧ	2.0 x 10°	150	(Ti,Al,) N	0.5	0.5	8 .1		8.8m (8.5m)	face
	L		الدرائية بالأدل بالتجارية والمتحددة								lapping
Invention	*	40	3.0 x 10 ⁻⁷	150	(T1,A1,)N	0.4	0.6	10.2	3.9	3.9m (3.5m)	
	in.	*0	0.5 x 10-7	150	(T1,A1,)N	0.5	0.5	6.0	yr car	2.0m (1.7m)	
	æ	30	2.0 x 10 ⁻³	150	(T1,A1,) N	0.5	0.5	15.4	2.5	4.2m (4.0m)	
	7	20	2.0 x 10 ⁻³	150	(T1,A1,)N	0.5	0.5	22.5	1.2	3.3m (3.3m)	
•	æ	60	0.5 x 10.7	150	(T1,A1,)N	0.5	0.5	0,8	6.1	1.0m (0.8m)	
	9	100	2.0 × 10.3	150	(Ti,Al,)N	0.5	0.5	0.7	5.5	0.9m (0.9m)	
Comparison	10	100	3.0 × 10.3	150	(Ti,Al,) N	0.5	0,5	0.9	4.8	0.8m (0.7m)	
	11	150	2.0 x 10 ⁻³	150	(Ti,Al,)N	0.5	0.5	0.2	7.2	0.1m (0.1m)	
	12	100	0.5 x 10 ⁻³	150	(Ti_Al,)N	0.4	0.6	0.1	6.8	0.2m (0.1m)	

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Examples 2:

The apparatus as used for coating according to Example 1 was also used for coating the samples Nr. 13 to 22 of table 2. The thickness of the overall coating was again 5 µm. It may be seen that in addition to the coating according to Example 1 there was applied an interlayer of titanium nitride between the MeX layer and the tool body and an outermost layer of the respective material as stated in table 2. The condition with respect to I(200) and average noise level, measured according to MS was largely fulfilled.

It may be noted that provision of the interlayer between the MeX layer and the tool body already resulted in a further improvement. An additional improvement was realised by providing an outermost layer of one of the materials titanium carbonitride, titanium aluminum oxinitride and especially with an outermost layer of aluminum oxide. Again, it may be seen that by realising the inventively stated Q_I values with respect to the comparison samples number 19 to 22, a significant improvement is realised.

20 The outermost layer of aluminum oxide of 0.5 μm thickness, was formed by plasma CVD.

The coated inserts of cemented carbide were tested under the same cutting conditions as those of Example 1, $Q_{\rm r}$ was measured according to MS.

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(Tabelle 2)

		:						
Sample No.		Inter-	rial Layer	×	>-	most	Z, = 1 1 (200) /	Attalnable cucting
1		layer				Layer	I (111)	+ill delamination)
		ium)						
	13	Tin	(TixAl,) N	0.5	0.5	1	1.5	4.5m (4.2)
		(0.4 um)	(4.6 µm)					
tropout	1.4	N,L	(Ti,Al,) N	0.5	0.5	Ticn	7.2	7.8 (7.5m)
31150513	1	(0, 4 um)	(4.1 µm)		1	(0.5 µm)		
Tayyoution	2		(Tial,)N	0.5	0.5	Tich	6.8	6.0m (5.5m)
TIMETER	}		(4.4 nm)			(0.5 µm)		
	,	4-	(#i.Al.) N	0.5	0.5	(Tial) NO		6.2m (6.0m)
	0		(m) (v)			(m.5.0)		
		(0.4 mm)	/m+ + - E \	1			13 6	10.1m (9.8m)
	17	TIN	(TixA)	0.5	0.5 0.5	******	C . 7 T	
		(0.4 µm)	(4.1 µm)			(0.5 µm)		(100 0)
	187		_	0.5	0.5		7.0	(NC. C) 110.2
			(4.1 um)			(0.5 µm)		
		_	\downarrow	2	0.5		0.8	1.5m (1.2m)
		MILT.	(TTXWTE) IN				8	1.9m (1.5m)
Comparison 20	20	TIN	(Tiral,) N	0.5	0.5 0.5			, on (1 Km)
4	5	N i di	(Ti.Al.)N	0.5	0.5 0.5	Ticn	7.0	(m) + ma + T
		$oldsymbol{\perp}$	Wi LA I'M	0.5	0.5 0.5	(TiAl)NO	0.1	0.6m (U.4m)
	7.7	LIN	1 4 TX TX 1 1 1		1			

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Example 3:

Again, cemented carbide inserts were coated with the apparatus of Example 1 with the MeX layer as stated in table 3, still fulfilling the Q conditions as inventively stated and, by far, 5 the condition of I(200) with respect to average noise level, measured according to MS. Thereby, there was introduced one of zirconium, hafnium, yttrium, silicon and chromium, with the amount as stated above, into Me.

The coated inserts were kept in an air oven at 750°C for 30 10 min. for oxidation. Thereafter, the resulting thickness of the oxide layer was measured. These results are also shown in table 3. For comparison, inserts coated inventively with different Me compounds of the MeX material were equally tested. It becomes evident that by adding any of the elements according to samples 23 to 32 to Me, the thickness of the resulting oxide film is significantly reduced. With respect to oxidation the best results were realised by adding silicon or yttrium.

It must be pointed out, that it is known to the skilled artisan, that for the MeX material wear resistant layers there is valid: The better the oxidation resistance and thus the thinner the resulting oxide film, the better the cutting performance.

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(Table 3)

	Comparison								A.S		T		Present	26	60	اد	24		32		sample No.	
35	34		₩ 	32		ω 	30		29 —	28	5	27				n	*	-				
(Ti,Al,Ta,)N	A Literature Co.	שו לעוות גדון	(Ti,Al,) N		ITT AL Y-Si) N	(Ti,Al,Hf,)N	1 2 2 20 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	AL PI SI-)N	(Ti,Al,Sl, N	1 - A - A	(T-1. A.) X	(TizAlyZE2) N		(Ti,Al,Y,)N		(TLALZra)N	(T3 _k AL _y Cx ₂) re	A. IN	(Ti,Al,Y,)N		Layer Composition	
					0.1																*	£
	0.4	0.4		0.4	0.3	0.4	•	0.48		→	0.4		0.25	0.43	20	0.48		0.48	0.40	,	;	×
	0.5	0.5	, - -	0.6	0.5		 Э	0.5			0.0	,	0.5		, r	0.5	۰ ۳	o.	0.0	o n		<
	0.1	5:	0		0.1	3	٠.	0.04	3	0.1		2	0.25	,	0.25		0 03	0.02		0.02		N
	3.3		2.5	1.8		0 - 05	0.9		n 2	0.1		0.00	0.0	3 m	0.1		0.7	0.8		0.7	Oxide Film (jum)	Thickness of

- 1.3 -

Example 4:

An apparatus and a coating method as used for the samples of Example 1 was again used.

HSS drills with a diameter of 6 mm were coated with a 4.5 μm 5 MeX and a TiN interlayer was provided between the MeX layer and the tool body, with a thickness of 0.1 $\mu\text{m}\,.$ The test condition were:

HSS twist drill, dia. 6 mm

DIN 1.2080 (AISI D3)

were:

Tool: HSS twist drill

Material: DIN 1.2080 (AIS

Cutting parameters: $v_c = 35 \text{ m/min}$

f = 0.12 mm/rev.

15 mm deep blind holes with coolant.

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(Table 4)

		Voltage (-V)	N ₃ . Pressure (mbar)	Arc current (A)	inter- layer layer		×	> -	ы	o ï	X Y z Q, Residual Number of Stress (3Pa) drilled holes	Number of drilled holes
	36	9	3.0 × 10.3	200	T-1 N	:	,	ŀ				
Present					0.1 um	TITY O'D WINTER		#		4	2.1	210
Invention	37	40	3.0 x 10.1	200	TIN	0 c co 0 1 0 55 0 21 a (4 14)	0 50	7.0	ç	6		
					0.1 00		?		Y	, ,	×.	130
	38	150	1.0 x 10"	200	TIN	4.0 9.0 M (4.47)	9.0	4		60.0	-	
Comparagon		T			0.1 mm		:	<u>ب</u>		2	e.	9
	30	150	1.0 x 10.	200	TIN	(TLALE,)N 0.58 0.4 0.02 0.1	0.58	4.0	0.02	0.1	4.4	á
					0.1 um					!		?

The lifetime of the tool was determined by the number of holes which could be drilled before failure of the drill.

The results of the inventively coated drills are shown as samples No. 36 and 37 in Table 4, the samples No. 38 and 39 again show comparison samples. Again, I(200) exceeded 20 times intensity average noise level by far, for samples 36, 37, as measured by MS.

Example 5:

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Again, the apparatus and method as mentioned for Example 1 was used for coating HSS roughing mills with a diameter of 12 mm with a 4.5 µm MeX layer. There was provided a titanium nitride interlayer with a thickness of 0.1 µm between the MeX layer and the tool body. The test conditions were:

HSS roughing mill, dia. 12 mm 15 Tool:

z = 4

Material: AISI H13 (DIN 1.2344)

640 N/mm²

Cutting parameters: $v_c = 47.8 \text{ m/min}$

20 0.07 mm

18 mm

6 mm

climb milling, dry.

The HSS roughing mill was used until an average width of flank wear of 0.2 mm was obtained.

Sample No. 40 in Table No. 5 shows the results of the inventively coated tool, sample 41 is again for comparison. Again, I(200) of sample Nr. 40 fulfilled the condition with respect to noise, as measured by MS.

s retearther treatment.

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(Table 5)

		Bias Voltage (-V)	las N. ; oltage Pressure -v) (mbar)	Arc current (A)	inter- layer layer		×	> -	ڻ ک	Residual Cutting Stress (GPa) distance (m)	Cutting distance (m)
Dragant	40	04	40 3.0 × 10.7	200	7.1K 0.1 µm	(Ti,Ai,) N 0.6 0.4 5.4	9.0	0.4	5.4	2.1	350
Invention											
Comparison	2	150	150 1.0 x 10. ²	200	TIN 0.1 µm	N(^tv*il)	9.	4	0.6 0.4 0.03	.	11m (chipping and pee-

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Example 6:

Again, the apparatus and coating method according to Example 1 was used. Solid carbide end mills with a diameter of 10 mm with 6 teeth were coated with a 3.0 μm MeX layer. There was provided a titanium nitride interlayer with a thickness of 0.08 µm between the MeX and the tool body. Test conditions for the end mills were:

Tool:

Solid carbide end mill, dia. 10 mm

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Material: 10

AISI D2 (DIN 1.2379)

60 HRC

Cutting parameters: $v_c = 20 \text{ m/min}$

 $f_e = 0.031 \text{ mm}$

15 mm

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= 1 mm

Climb milling, dry

The solid carbide end mills were used until an average width of flank wear of 0.20 mm was obtained. It is to be noted that solid carbide end mills do not belong to that group of tool 20 which is inventively coated with a hard material layer having $Q_r \ge 1$. From the result in Table 6 it may clearly be seen that for this kind of tools Q, > 1 does not lead to an improvement. Again, the I(200) to noise condition, measured with MS, was fulfilled for sample No. 42, for sample No. 43 the I(111) to noise condition was fulfilled.

(Table 6)

		Bias Voltage [-V]	Blas Ny. Voltage Pressure (-V) (mbar)	Arc ourrent (A)	inter- layer layer		×	*	đ	x y Q, Residual Cutting Stress (GPa) distance (m)	Cutting distance (m)
Present	42	40	40 3.0 × 10.1	200	T11N 0.08 µm	(Ti_Aly)N 0.6 0.4 5.0 3.2	9.0	4.0	5.0	3.2	17m
Invention											
Comparison	43	1.50	150 1.0 x 10.7 200	203	N17 0.08 µm	(Ti.Al) 0.6 0.4 0.05 4.7	9.6	0.4	0.05	4.7	32m

Example 7:

Again, an apparatus and method as used for the samples of Example 1 were used.

Solid carbide drills with a diameter of 11.8 mm were coated with a 4.5 μ m MeX layer. There was provided a TiN interlayer between the MeX layer and the tool body.

Test conditions:

Tool:

Solid carbide drill, diam. 11.8 mm

Workpiece:

Cast iron GG25

Machining conditions: v_c = 110 m/min

f = 0.4 mm/rev.

Blind hole 3 x diam.

No coolant

The solid carbide drills were used until a maximum width of flank wear of 0.8 mm was obtained. The I(200) to noise condition was again fulfilled, measured with MS.

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(Table 7)

		Bias Voltage (-V)	Bias N. ' Voltage Pressure (-V) (mbar)	Arc current (A)	Inter- layer	layer	×	- δ		Residual Drilling Stress (GPa) distance (m)	Drilling distance (m)
	1.	0+	3.0 × 10.3	200	TiN 0.3 um	MI,LALT)	9.0	4 :0	0.6 0:4 5.4	2.1	##56
Invention											
	45	150	150 1.0 × 10.2	200	TIN 0.1 LM	(Tiral,) N	9.0	4 .0	0.6 0.4 0.03	4.5	48.5m

Example 8:

Again, the apparatus and method as stated in Example 1 were used.

Cemented carbide inserts for turning with a shape in accordance with CNGP432 were coated with a 4.8 µm MeX layer. There was provided a TiN interlayer with a thickness of 0.12 µm between the MeX layer and the tool body. The test conditions were:

Tool:

Carbide insert (CNGP432)

Material:

DIN 1.4306 (X2CrNi 1911)

Cutting parameters:

 $v_c = 244 \text{ m/min}$

 $f = 0.22 \, mm/rev.$

 $a_n = 1.5 \text{ mm}$

with emulsion

The tool life was evaluated in minutes. The indicated value is an average of three measurements. Again, I(200)/noise condition, measured with MS, was fulfilled.

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(Table 8)

		Blas Voltage (-V)	Blas M. Voltage Pressure (-V)	Arc Current (A)	inter- layer	layer	> *	<u>ب</u>	ď	Residual Tool life Stress (GPa) (min)	Tool life (min)
Present	46	40	3.0 × 10-3	200	rin 0.12 um	(Tial,) N	0.6 0.4 5.B	4.0	5.8	1.9	19.1 min
Invention									Ī		
Compartson	47	150	.50 1.0 x 10.4	200	Tin 0.12 um	(Ti,Al,) N	0.6 0.4 0.04	9.0	0.04	4.9	S.5 min
									T		

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In Fig. 1 there is shown, with linear scaling a diagram of nitrogen partial pressure versus bias voltage of the tool body as applied for reactive cathodic arc evaporation as the reactive PVD deposition method used to realise the Examples which were discussed above.

All the process parameters of the cathodic arc evaporation process, namely

- arc current;
- process temperature;
 - deposition rate;
 - evaporated material;
 - strength and configuration of magnetic field adjacent the arc source;
- 15 geometry and dimensions of the process chamber and of the workpiece tool to be treated

were kept constant. The remaining process parameters, namely partial pressure of the reactive gas - or total pressure - and bias voltage of the tool body to be coated as a workpiece and with respect to a predetermined electrical reference potential, as to the ground potential of the chamber wall, were varied.

Thereby, titanium aluminum nitride was deposited. With respect to reactive gas partial pressure and bias voltage of the tool body, different working points were established and the result-

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ing \mathbf{Q}_{I} values at the deposited hard material layers were measured according to MS.

It turned out that there exists in the diagram according to fig. 1 an area P, which extends in a first approximation linearly from at least adjacent the origin of the diagram coordinates, wherein the resulting layer leads to very low XRD intensity values of I(200) and I(111). It is clear that for exactly determining the limits of P, a high number of measurements will have to be done. Therein, none of the I(200) and I(111) intensity values is as large as 20 times the average noise level, measured according to MS.

On one side of this area P and as shown in fig. 1 Q_I is larger than 1, in the other area with respect to P, Q_I is lower than 1. In both these areas at least one of the values I(200), I(111) is larger than 20 times the average noise level, measured according to MS.

As shown with the arrows in fig. 1, diminishing of the partial pressure of the reactive gas - or of the total pressure if it is practically equal to the said partial pressure - and/or increasing of the bias voltage of the tool body being coated, leads to reduction of Q_i. Thus, the inventive method for producing a tool which comprises a tool body and a wear resistant layer system, which latter comprises at least one hard material layer, comprises the steps of reactive PVD depositing the at least one hard material layer in a vacuum chamber, thereby preselecting process parameter values for the PVD deposition process step beside of either or both of the two process parameters, namely of partial pressure of the reactive gas and of bias voltage of the tool body. It is one of these two parame-

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ters or both which are them adjusted for realising the desired Q, values, thus, and according to the present invention, bias voltage is reduced and/or partial reactive gas pressure is increased to get Q_i values, which are, as explained above, at

- least larger than 1, preferably at least larger than 2 or even 5 and even better of 10. Beside the inventively exploited Q_{τ} value, in this "left hand" area, with respect to P, I(200) is larger, mostly much larger than 20 times the average noise level of intensity, measured according to MS.
- In fig. 2 a typical intensity versus angle 2θ diagram is shown for the titanium aluminum nitride hard material layer deposited in the $Q_{\rm r} \ge 1$ region according to the present invention of fig. 1, resulting in a Q value of 5.4. The average noise level N* is much less than I(200)/20. Measurement is done according to MS.
- In Fig. 3 a diagram in analogy of that in fig. 2 is shown, but the titanium aluminum nitride deposition being controlled by bias voltage and nitrogen partial pressure to result in a $Q_{\rm I} \le$ 1. The resulting $Q_{\scriptscriptstyle T}$ value is 0.03. Here the I(111) value is larger than the average noise level of intensity, measured according to MS.
- Please note that in fig. 1 the respective $Q_{\scriptscriptstyle \rm I}$ values in the respective regions are noted at each working point measured (according to MS).
- In fig. 4 a diagram in analogy to that of the figs. 2 and 3 is 25 shown for working point P, of fig. 1. It may be seen that the intensities I(200) and I(111) are significantly reduced compared with those in the area outside P. None of the values

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I(200) and I(111) reaches the value of 20 times the noise average level N^{\pm} .

Thus, by simply adjusting at least one of the two Q_r -controlling reactive PVD process parameters, namely of reactive gas partial pressure and of workpiece bias voltage, the inventively exploited Q_r value is controlled.

In fig. 1 there is generically shown with $\partial Q_r < 0$ the adjusting direction for lowering Q_T , and it is obvious that in opposite direction of adjusting the two controlling process parameters, and increase of Q_T is reached.